FORM PTO-1390 (REV 10-94) U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER: 11318.58USWO

U S APPLICATION NO (If known, see 37 C F R 1 5): Unknown 09/914551

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/DE00/00485	February 22, 2000	March 5, 1999

TITLE OF INVENTION

METHOD FOR CONTROLLING MICROORGANISMS IN A SUGAR-CONTAINING AQUEOUS PROCESS MEDIUM

APPLICANT(S) FOR DO/EO/US

MAYE et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. [X] This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. [ ] This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. [X] This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).
4. [X] A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. [X] A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. [X] is transmitted herewith (required only if not transmitted by the International Bureau).
   b. [X] has been transmitted by the International Bureau.
   c. [ ] is not required, as the application was filed in the United States Receiving Office (RO/US)
6. [X] A translation of the International Application into English (35 U.S.C. 371(c)(2)); Certification of Translation.
7. [X] Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. [ ] are transmitted herewith (required only if not transmitted by the International Bureau).
   b. [ ] have been transmitted by the International Bureau.
   c. [ ] have not been made; however, the time limit for making such amendments has NOT expired.
   d. [X] have not been made and will not be made.
8. [X] A translation of the amendments.
9. [X] An unsigned oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. [ ] A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. [ ] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. [ ] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. [X] A first preliminary amendment; Marked-up version of claims; Abstract.
    [ ] A SECOND of SUBSEQUENT preliminary amendment.
14. [ ] A substitute specification.
15. [ ] A change of power of attorney and/or address letter.
16. [X] Other items or information: ISR/ISA/210 in German and its English translation; Amended claims in German and its English translation

U.S. APPLICATION NO (If known, see 37 C F R 1 5)	INTERNATIONAL APPLICATION NO	ATTORNEY'S DOCKET NUMBER
Unknown 09/914551	PCT/DE00/00485	11318.58USWO

17. [X] The following fees are submitted:

CALCULATIONS PTO USE ONLY

BASIC NATIONAL FEE (37 CFR 1.492(a) (1)-(5)):

Search Report has been prepared by the EPO or JPO **$860.00**

International preliminary examination fee paid to USPTO (37 CFR 1.492(a)(1)) **$690.00**

No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) **$710.00**

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(3)) paid to USPTO **$1000.00**

International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) **$100.00**

ENTER APPROPRIATE BASIC FEE AMOUNT =				**$860.00**	
Surcharge of **$130.00** for furnishing the oath or declaration later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				**$**	
CLAIMS	NUMBER FILED		NUMBER EXTRA	RATE	
Total claims	21	-20 =	1	X **$18.00**	**$18.00**
Independent claims	1	-3 =	0	X **$80.00**	**$**
MULTIPLE DEPENDENT CLAIM(S) (if applicable)				+ **$260.00**	**$**
TOTAL OF ABOVE CALCULATIONS =					**$878.00**
Reduction by 1/2 for filing by small entity, if applicable. Small entity status is claimed pursuant to 37 CFR 1.27					**$**
SUBTOTAL =					**$878.00**
Processing fee of **$130.00** for furnishing the English translation later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(f).				+	**$**
TOTAL NATIONAL FEE =					**$878.00**
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property				+	**$**
TOTAL FEES ENCLOSED =					**$878.00**
				Amount to be: refunded	**$**
				charged	**$**

a. [X] Check(s) in the amount of $878.00 to cover the above fees is enclosed.

b. [ ] Please charge my Deposit Account No. _____ in the amount of $ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. [X] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-2725.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO
John J. Gresens
MERCHANT & GOULD
P.O. Box 2903
Minneapolis, MN 55402-0903

SIGNATURE: John J. Gresens

NAME: John J. Gresens

REGISTRATION NUMBER: 33,112

S/N unknown

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	MAYE et al.	Serial No.:	unknown
Filed:	concurrent herewith	Docket No.:	11318.58USWO
Title:	METHOD FOR CONTROLLING MICROORGANISMS IN A SUGAR-CONTAINING, AQUEOUS PROCESS MEDIUM		

CERTIFICATE UNDER 37 CFR 1.10
'Express Mail' mailing label number: EL669944085US
Date of Deposit: August 29, 2001
I hereby certify that this correspondence is being deposited with the United States Postal Service 'Express Mail Post Office To Addressee' service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner for Patents, Washington, D.C . 20231.

By: Omesh Singh
Name: Omesh Singh

PRELIMINARY AMENDMENT

Box PCT
Assistant Commissioner for Patents
Washington, D. C. 20231

Dear Sir:

In connection with the above-identified application filed herewith, please enter the following preliminary amendments, based on claims amended in prosecution of the international application, a copy of which is enclosed herewith:

IN THE ABSTRACT

Insert the attached Abstract page into the application as the last page thereof.

IN THE SPECIFICATION

A courtesy copy of the present specification is enclosed herewith. However, the World Intellectual Property Office (WIPO) copy should be relied upon if it is already in the U.S. Patent Office.

IN THE CLAIMS

Please amend claims 1-7, 9, 12-15 and 17-21 as follows:

1. (amended) Procedure for control of the content of microorganisms in a sugary, aqueous process medium of extraction systems of the sugar industry using hops acid as the active substance,

characterized by the fact that

hops acid brought into solution in an aqueous alkaline medium is added to the process medium, whereby the pH value of the added solution is higher than the pH value of the process medium (and) the hops acid in the process medium passes over from the dissociated form into the dissociated form.

2. Procedure according to claim 1,

characterized by the fact that

addition of the solution to the process medium is done in discontinuous manner.

3. Procedure according to claim 1,

characterized by the fact that

the solution displays hops acid in a concentration of 2 – 40%, preferably 5- 20%, preferably 10 – 15%.

4. (amended) Procedure according to claim 1,

characterized by the fact that

the solution added to the process medium displays a pH value of 7.0 – 13.0, preferably 7.5 – 12.0, preferably 9.5 – 11.0.

5. (amended) Procedure according to claim 1,

characterized by the fact that

being dealt with – at least predominantly – in the case of hops acid is β-acid.

6. (amended) Procedure according to claim 1,

characterized by the fact that

being dealt with – at least predominantly – in the case of hops acid is α-acid and/or iso-α-acid.

7. (amended) Procedure according to claim 1,

characterized by the fact that

in the case of the hops acid being dealt with – at least predominantly – is isomerized hops acid and/or its derivatives, or in any event a mixture thereof.

9. (amended) Procedure according to claim 1,

characterized by the fact that

provided as an alkaline medium is an alkaline hydroxide, in particular potassium hydroxide or sodium hydroxide, or a mixture thereof.

12. (amended) Procedure according to claim 1,

characterized by the fact that

the hops acid is dissolved in the alkaline medium as salt.

13. (amended) Procedure according to claim 1,

characterized by the fact that

the solution is added to the process medium manually.

14. (amended) Procedure according to claim 1,

characterized by the fact that

the solution is added to the process medium over already available dosing systems.

15. (amended) Procedure for the production of a solution of hops acid for addition to a sugary, aqueous process medium, in particular of the sugar industry according to the procedure based on claim 1,

the following procedural steps comprising:

a) preparation of an aqueous medium;

b) heating;

c) addition of hops acid, in particular melted hops acid, measuring the amount of hops acid such that the end concentration lies within a prescribed concentration range;

d) addition of the alkaline medium for reaching a predetermined pH value;

e) mixing the alkaline medium with the added-in hops acid;

f) maintaining the mixture at an elevated temperature over a prescribed period of time;

g) separating out the hops acid solution from the mixture or vice-versa, as well as

h) cooling the hops acid solution.

17. (amended) Procedure according to claim 15,

characterized by the fact that

the mixture is held at a temperature in the range of 40 – 80° C, preferably 60 – 80° C, preferably 65 – 75° C.

18. (amended) Procedure according to the foregoing claim 15,

characterized by the fact that

hops acid solution is cooled down to a temperature below 10° C, preferably to a temperature in a range from 2 – 7° C.

19. (amended) Procedure according to one of the foregoing claim 15,

characterized by the fact that

the separated out solution of hops acid displays a pH value in the range of 7.0 – 13.0, preferably 7.5 – 12.0, preferably 9.5 – 11.0.

20. (amended) Procedure according to claim 15,

characterized by the fact that

used as hops acids are β-acids, α-acids, iso-α-acids or a mixture thereof, or isomerized hops acids and/or their derivatives, in particular –at least predominantly – tetrahydro-α-acid (THAA) or hexahydro-β-acid (HHBA) or iso-α-acid (IAA), rho-iso-α-acid (RIAA), tetrahydro-iso-α-acid (THIAA) and/or hexahydroxide-iso-α-acid, or a mixture thereof.

21. (amended) Use of hops acids for combating microorganisms in a sugary, aqueous process medium, in particular of the sugar industry,

characterized by the fact that

hops acid brought into an alkaline solution is added to the process medium, whereby the pH value of the solution is higher than the pH value of the process medium, and the hops acid in the process medium passes over from the dissociated form into the non-dissociated form based on claim 1.

REMARKS

The above preliminary amendment is made to remove multiple dependencies from claims 1-7, 9, 12-15 and 17-21.

A new abstract page is supplied to conform to that appearing on the publication page of the WIPO application, but the new Abstract is typed on a separate page as required by U.S. practice.

Applicants respectfully request that the preliminary amendment described herein be entered into the record prior to calculation of the filing fee and prior to examination and consideration of the above-identified application.

If a telephone conference would be helpful in resolving any issues concerning this communication, please contact Applicants' primary attorney-of record, John J. Gresens (Reg. No. 33,112), at 612.371.5265.

Respectfully submitted,

MERCHANT & GOULD P.C.
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Dated: August 29, 2001

By _____
John J. Gresens
Reg. No. 33,112

JJGRESENS/kas

Marked-up Copy of Claims

1. [Procedure for combating microorganisms in a sugary, aqueous process medium, in particular of the sugar industry inserting hops acid as the active substance,]

"Procedure for control of the content of microorganisms in a sugary, aqueous process medium of extraction systems of the sugar industry using hops acid as the active substance,"

characterized by the fact that

[hops acid in an aqueous alkaline medium is added in solution to the process medium, whereby the pH value of the added solution is higher than the pH value of the process medium; the hops acid in the process medium passes over from the dissociated form into the non-dissociated form.]

"hops acid brought into solution in an aqueous alkaline medium is added to the process medium, whereby the pH value of the added solution is higher than the pH value of the process medium (and) the hops acid in the process medium passes over from the dissociated form into the dissociated form."

2. Procedure according to [one of the foregoing claims] "claim 1",

characterized by the fact that

addition of the solution to the process medium is done in discontinuous manner.

3. Procedure according to [Claim 1 or 2] "claim 1",

characterized by the fact that

the solution displays hops acid in a concentration of 2 – 40%, preferably 5-20%, preferably 10 – 15%.

4. Procedure according to [one of the forgoing claims] "claim 1",

characterized by the fact that

the solution added to the process medium displays a pH value of 7.0 – 13.0, preferably 7.5 – 12.0, preferably 9.5 – 11.0.

5. Procedure according to [one of the foregoing claims] "claim 1",

characterized by the fact that

being dealt with – at least predominantly – in the case of hops acid is β-acid.

6. Procedure according to [one of Claims 1 –4] "claim 1",

characterized by the fact that

being dealt with – at least predominantly – in the case of hops acid is α-acid and/or iso-α-acid.

7. Procedure according to [one of Claims 1-6] "claim 1",

characterized by the fact that

in the case of the hops acid being dealt with – at least predominantly – is isomerized hops acid and/or its derivatives, or in any event a mixture thereof.

9. Procedure according to [one of the foregoing claims] "claim 1",

characterized by the fact that

provided as an alkaline medium is an alkaline hydroxide, in particular potassium hydroxide or sodium hydroxide, or a mixture thereof.

12. Procedure according to [one of the foregoing claims] "claim 1",

characterized by the fact that

the hops acid is dissolved in the alkaline medium as salt.

13. Procedure according to [one of the foregoing claims] "claim 1",

characterized by the fact that

the solution is added to the process medium manually.

14. Procedure according to [one of the foregoing claims 1 –12] "claim 1",

characterized by the fact that

the solution is added to the process medium over already available dosing systems.

15. Procedure for the production of a solution of hops acid for addition to a sugary, aqueous process medium, in particular of the sugar industry according to the procedure based on [the foregoing claims 1 – 14] "claim 1",

the following procedural steps comprising:

a) preparation of an aqueous medium;

b) heating;

c) addition of hops acid, in particular melted hops acid, measuring the amount of hops acid such that the end concentration lies within a prescribed concentration range;

d) addition of the alkaline medium for reaching a predetermined pH value;
e) mixing the alkaline medium with the added-in hops acid;
f) maintaining the mixture at an elevated temperature over a prescribed period of time;
g) separating out the hops acid solution from the mixture or vice-versa, as well as
h) cooling the hops acid solution.

17. Procedure according to [Claim 15 or 16] "claim 15",

characterized by the fact that

the mixture is held at a temperature in the range of 40 – 80° C, preferably 60 – 80° C, preferably 65 – 75° C.

18. Procedure according to the foregoing [claims 15 – 17] "claim 15",

characterized by the fact that

hops acid solution is cooled down to a temperature below 10° C, preferably to a temperature in a range from 2 – 7° C.

19. Procedure according to one of the foregoing [Claims 15 – 18] "claim 15",

characterized by the fact that

the separated out solution of hops acid displays a pH value in the range of 7.0 – 13.0, preferably 7.5 – 12.0, preferably 9.5 – 11.0.

20. Procedure according to [one of the foregoing claims 15 – 19] "claim 15",

characterized by the fact that

used as hops acids are β-acids, α-acids, iso-α-acids or a mixture thereof, or isomerized hops acids and/or their derivatives, in particular –at least predominantly – tetrahydro-α-acid (THAA) or hexahydro-β-acid (HHBA) or iso-α-acid (IAA), rho-iso-α-acid (RIAA), tetrahydro-iso-α-acid (THIAA) and/or hexahydroxide-iso-α-acid, or a mixture thereof.

21. Use of hops acids for combating microorganisms in a sugary, aqueous process medium, in particular of the sugar industry,

characterized by the fact that

hops acid brought into an alkaline solution is added to the process medium, whereby the pH value of the solution is higher than the pH value of the process medium, and the hops acid in the process medium passes over from the dissociated form into the non-dissociated form based on [one of the Claims 1 – 14] "claim 1".

1/prts

METHOD FOR CONTROLLING MICROORGANISMS IN A SUGAR-CONTAINING, AQUEOUS PROCESS MEDIUM

DESCRIPTION

This invention concerns a procedure for combating microorganisms in a sugary, aqueous process medium, especially the sugar industry using hops acid as the active substance. The invention further concerns a method for the production of a solution of hops acid for use in the aforementioned procedure, as well as use of the hops acid for combating microorganisms in a sugary, aqueous process medium, in particular of the sugar industry.

The bacteriostatic action of hops acid has been known for a long time. Hops acids were used for many years for the preservation of beer. The use of hops acid in brewing has, however, retreated into the background through bacteria-free fermentation and racking.

Known from EP 0 681 029 A2 is a procedure for inhibiting thermophilic microorganisms in the presence of sugary, aqueous mediums, where a hops base additive, preferably hops extract, is furnished in liquid or emulsified form to the sugary, aqueous mediums of the sugar industry (i.e. extracts of sugar-containing plants), and is worked in at temperatures between 50° C and 80° C takes place. The solution of the hops acid extract is done in water, however also with addition of alcohol. Supplying the dissolved or emulsified hops product can be done continuously or discontinuously (shock dosing)

Already known from US 5,286,506 is how to use hops acid for combating bacteria, in particular listeria in processed food products. Its use follows hereby due to the fact that solid processed food products are immersed in a solution of β-acid or sprayed with it.

Known from Arch. Mikrobiol. 94 (1973, pp. 159-171 is that hops acids in the range of a minimal concentration act bacteriostatically, however at higher concentrations act toxically. In the case of use of so-called "β-acids" as a special type of the hops acids, which according to the aforementioned publication show the highest bacteriostatic action in comparison to α-acid as well as iso-α-acids, because of low solubility certain concentrations of β-acid can not be exceeded.

At the earliest, still while combating thermophilic microorganisms, according to EP 0 681 029 A2, achieved by the prevailing high temperatures of the process medium there is a high solubility of β-acids and, therewith, a better efficiency.

The object of this invention consists of increasing the efficiency of the generic process.

The task is resolved in accord with the invention in that hops acid brought into solution in an aqueous alkaline medium is added to the process medium, whereby the pH value of the added-in solution is higher than the pH value of the process medium, and the hops acid in the process medium passes over from the dissociated form into the non-dissociated form. Because of the small dosage amount in added solution in comparison to the process medium, the solution after addition into the process medium assumes completely the pH value of the process medium, whereby the hops acid passes over from the dissociated form into the non-dissociated, antibacterially active form. Astonishingly, it has been shown that the action of hops acid, when in the process medium it passes from the dissociated condition into the non-dissociated form, is particularly good. This has as a consequence that, for obtaining the desired action, the total insertion of hops acid can be greatly reduced in comparison to earlier times. On the other hand, also achievable is an increased action with the same dosage as earlier. The procedure according to the invention is used in the case of process mediums, in particular in the form of sugary plant extraction solutions of the sugar producing process.

Practically, the solution is added to the process medium periodically, i.e. an addition occurs at certain points in time within a short period of time, when local and short term high concentrations are adjusted (shock dosing). A dosing of this kind by means of the high local concentrations works against an adaptation of microorganisms,

Practically, the solution to be supplied to the process medium displays hops acid in a concentration of 2 – 40%, preferably 5 – 20%, particularly preferable 10 – 15%. High concentrations are particularly favorable from the point of view of temporary storage as well as with regard to transport.

The pH value of the solution added to the process medium lies in a range of 7.0 – 13.0, preferably 7.5 – 11.5, preferably 9.5 – 10.5. In this range, a particularly high efficiency is reached by use of this solution. The solution can be added without the danger of caustic action on the human skin. Moreover, in contrast to other chemical agents, the solution develops no unpleasant or health-endangering vapor pressure.

A particularly high efficiency results with the use of β-acids as hops acid. However, α-acids or a mixture of α-acids and β-acids can also find application. In the production of the solution, α-acids are converted into iso-α-acids, and as such retain their bacteriostatic action.

According to the invention, in the case of the hops acid – at least predominantly – we can be dealing with isomerized hops acids and/or with their derivatives, or a mixture thereof. Here, practically, we are dealing with tetrahydro-α-acid (THAA), or with hexahydro-β-acid (HHBA), and in the case of the isomerized hops acid derivatives with iso-α-acid (IAA), rho-iso-α-acid (RIAA), tetrahydro-iso-α-acid (THIAA) and/or hexahydroxide-α-acid or a mixture of the above compounds.

Practically, to be provided as an alkaline medium is an alkaline hydroxide, in particular potassium hydroxide and/or sodium hydroxide, or a mixture thereof. Practically, the concentration of the alkaline medium amounts to 0.1 – 5%, preferably 1 –4%, preferably 2 – 3%.

A special variant of the procedure according to the invention is characterized by the fact that, besides addition of hops acid brought into solution, additionally supplied to the process medium is alkali liquor (lye), preferably in concentrations of 5 – 25%; undertaken thereby is a treatment under reinforced alkaline conditions. The reinforced alkaline conditions in the process medium take care of a delayed precipitation of the β-acids and, therewith, an additional increase in efficiency. This increase in efficiency works out particularly favorably in the case of discontinuous addition of the solution.

The solution, in particular in the case of the aforementioned range, can be supplied by manually pouring in, e.g. using the trough extraction system.

Alternatively, in the case of closed dosing systems, which are available in many sugar factories for emission-free dosing of formalin, the solution can be supplied over these dosing systems, i.e. combating of microorganisms can be undertaken while retaining the already existing procedural technology (closed dosing systems). The invention further concerns a procedure for producing a solution of hops acid for use in the procedure in accordance with Claims 1 – 14, which comprise the following steps:

a) preparation of an aqueous medium;
b) heating;
c) addition of hops acid, in particular melted hops acid, with measurement of the amount of hops acid such that the end concentration lies within a prescribed concentration range;
d) addition of the alkaline medium for reaching a predetermined pH value;
e) mixing of the alkaline medium with the added hops acid;
f) maintaining the mixture in a high temperature range over a prescribed period of time;
g) separation of the hops acid solution from the mixture or vice-versa, as well as
h) cooling the hops acid solution.

Through means of the above procedure, a solution can be prepared which, at high concentrations of hops acid, can be temporarily stored and/or transported. At the same time, the solution guarantees a reduction of the overall insertion amount of hops acid, in comparison to earlier procedures. The procedural steps can be changed in their sequence. The aforementioned sequence guarantees a very exact setting of the pH value of the solution.

Practical arrangements of the procedure according to the invention based on patent Claim 13 are located in the other patent Claims 14 – 18.

Further claimed in a collateral manner is the use of hops acid for combating microorganisms in a sugary, aqueous process medium of the sugar industry, which is characterized in such a way that hops acid brought into solution in an alkaline medium is added to the process medium, whereby the pH value of the solution is higher than the pH value of the process medium, and the hops acid in the process medium passes over from the dissociated to the non-dissociated form in accordance with procedural Claims 1 -12.

The single drawing figure shows in a strongly simplified schematic sequence of the process the individual steps for execution of the procedure according to the invention.

For this purpose, one heats an aqueous solution to 70° C – 75° C and brings into this solution melted β-acid-containing hops extract. The amount of hops extract is measured such that the end concentration of the acid in solution should lie at about 10 – 15%, whereby higher concentrations of β-acid are particularly favorable from the point of view of a temporary storage or a long transport. Potassium hydroxide is added until the predetermined pH value is reached.

The mixture is then maintained at temperature about 15 – 30 minutes.

The mixture separates into clear, alkaline β-acid solution along with turbid, oily components. The clear, alkaline β-acid solution with a pH value of preferably bout 10 –10.5, is drawn off from the mixture and cooled to a temperature below room temperature, preferably 2 – 7° C. Next, it is supplied to the process medium in discontinuous fashion, i.e. shock dosing.

There, the solution mixes with the slightly acidic or at least less alkalinically- reacting process medium, whereby, because of the low dosing amount of highly concentrated β-acid solution, the mixture nearly completely assumes the pH value of the process stream, whereupon the β-acid passes over from its dissociated salt form into the non-dissociated, antibacterially active form.

The α-acids contained in the hops extract are converted during the production of the solution into iso-α-acids, and as such retain a bacteriostatic action.

A solution of this kind, because of a moderated alkalinity, displays favorable properties relative to transport, handling and temporary storage, and is stable over several months. Because of its composition, the solution can, for example, be dosed (measured) into trough extraction facilities of the sugar industry by manual pouring into hatches. There is neither a caustic action on human skin to be feared, nor does the alkaline solution, in comparison to other chemical agents, develop an unpleasant or health-endangering vapor pressure (as is the case with formalin). Likewise, because of the selected pH value for the solution, achievable with direct use of the solution is a strong increase in efficiency.

The solution can also be supplied through closed dosing systems that are available in many sugar factories for emission-free dosing of formalin, if the formalin pump is operated with soft water instead of formalin, and the alkaline hops acid solution is dosed into the suction line of the running pump. The alkaline solution can, hereby, be sucked in, pressed in by static height or by means of a second pump, whereby achieved by a short overrun of the water pump is a scavenging of the line.

When using the closed dosing system, the solution can be measured in, even under reinforced alkaline conditions by the additional use of alkali liquor (lye). In doing this, in parallel to the hops acid solution, measured into the process medium is lye in concentrations of 5 – 25%. Here in the case of temporary storage that would lead to β-acid losses, stronger alkaline conditions can also be selected for a short period of time. By supplementary use of lye and build up of alkaline striae in the process medium, achieved will be an at least slightly delayed precipitation or, to be precise, formation of the non-dissociated form of the β-acids and an additional improvement effect.

Finally, it is possible to start out right at the process medium, i.e. in the factory, from melted, commercially available hops extracts and, shortly before a shock dosing, to mix this with lye at elevated temperature. After a short solution time the entire mixture is measured in as a single shock dose. Also in doing this, short-time, stronger alkaline conditions can be selected, which in the case of a temporary storage would lead to losses in hops acid.

The process can be automated by time-control of the dosing pump and valves. Also, in this case the increase in efficiency that is the object of this invention also enters in.

Through means of the improved action, the total insertion of active substances is reduced, which is associated with various advantages. Either reduced costs result through reduced dosing or there is an increased action with the same dosing.

For hops products with the same concentration, transport volume is reduced by the increase in efficiency. Of further significance is that the solid residues of a sugar extraction are turned into feed, and in the case of extreme increase, the dose for combating partially adapted microorganisms of the products of oxidation of β-acids could lead to a bitter taste for feed. With an increase in efficiency, this disadvantage is reduced.

Dependent upon the environment and the legal situation, sugar factories have different optimal conditions for operation of the extraction system. In many cases, microorganisms in the lower concentration range are knowingly allowed in order to improve ability to express the extracted waste. In these types of factories, microorganism growth can be better limited by alkaline hops solutions. Other factories wish to suppress as completely as possible microorganism growth in the extraction system, in order to minimize sugar losses. Here also, an increase in the efficiency means a decreased insertion of active substances, and therewith a cost advantage.

The following examples take into account different arrangements in sugar factories.

Example 1

A 40% solution of potassium hydroxide (30 kg) is added to a stirred solution of beta fraction (200 kg, containing 55% β-acids) along with water (900 liters) at 70° C until a pH value of 10.5 is set. After a stirring period of two hours, the oil as well as the aqueous layers are allowed to separate. The aqueous layer is drawn off and cooled to 5°. Precipitates are removed in order to obtain an aqueous β-acid solution (1,000 liters) that is used in a sugar factory in an extraction tower, and with a processing capacity of 10,000 tons of beets per day. The existing formalin dosing facility is operated with soft water instead of formalin, and the alkaline solution is dosed into the suction line of the running formalin pump. For freely scavenging the line, post-rinsing for one minute is done with water. Dosing is done at three locations of the extraction stream, six times daily, with 17 liters of solution that corresponds in total to 31 g/ton of beets. With this dosage, the lactic acid content of the raw juice is limited to a value of 450 mg/kg, which is non-imparing to the pressibility of the waste.

Example 2

Produced is a solution in accordance with Example 1, which, analogous to Example 1, however, is dosed with the additional use of sodium lye. During dosing of 14 liters of alkaline

solution/dosing location, dosed at the same are 40 liters of 5% sodium lye, so that the alkaline conditions in the transport water stream, and during entrance into the juice stream, will be reinforced. Through means of the reinforced alkaline conditions, the desired effect is already reached at 25 g/tons of beets.

Example 3

Produced is a solution in accordance with Example 1 and used in a sugar factory, with a DDS extraction system and a processing capacity of 10,000 tons of beets/day for combating microorganism activity, whereby no targeted fermentation should be allowed. The solution is dosed in by manually pouring into the pressurized water circuit and into the hatches 2 and 3 of the extraction system. Because of the manual handling, use of additional lye is rejected. Six times per day 11 liters are brought to the mentioned locations; this corresponds in total to 20 g/ton of beets. When a first return of microorganisms is recognized from the nitrite or lactic acid conditions, dosing is done once at an earlier point in time.

Example 4

Available in a sugar factory with a processing capacity of 10,000 tons of beets/day, are contrivances for melting base extract and a vessel that can be temperature stabilized at 70° C. Lactic acid formation is to be limited in accordance with Example 1, whereby individual impacts are produced at various locations of the extraction system, time delayed at least 30 minutes. A half hour before an impact-dosing time, 20 liters of 70° C warm water, 6 liters of 11% sodium lye and 3.5 liters of base extract are mixed and stirred up until the impact-dosing point in time. Next, the solution is measured in and the container therewith becomes free for preparation of the next batch.

[t.n.: "amended claims" handwritten here]

AMENDED PATENT CLAIMS

1. Procedure for control of the content of microorganisms in a sugary, aqueous process medium of extraction systems of the sugar industry using hops acid as the active substance,

characterized by the fact that

hops acid brought into solution in an aqueous alkaline medium is added to the process medium, whereby the pH value of the added solution is higher than the pH value of the process medium (and) the hops acid in the process medium passes over from the dissociated form into the dissociated form

2. Procedure according to one of the foregoing claims, *[t.n.: sic]*

characterized by the fact that

addition of the solution to the process medium is done in discontinuous manner.

3. Procedure according to Claim 1 or 2,

characterized by the fact that

the solution displays hops acid in a concentration of 2 – 40%, preferably 5- 20%, preferably 10 – 15%.

4. Procedure according to one of the forgoing claims,

characterized by the fact that

the solution added to the process medium displays a pH value of 7.0 – 13.0, preferably 7.5 – 12.0, preferably 9.5 – 11.0.

5. Procedure according to one of the foregoing claims,

characterized by the fact that

being dealt with – at least predominantly – in the case of hops acid is β-acid.

6. Procedure according to one of Claims 1 –4,

characterized by the fact that

being dealt with – at least predominantly – in the case of hops acid is α-acid and/or iso-α-acid.

7. Procedure according to one of Claims 1-6,

characterized by the fact that

in the case of the hops acid being dealt with – at least predominantly – is isomerized hops acid and/or its derivatives, or in any event a mixture thereof.

8. Procedure according to Claim 7,

characterized by the fact that

in the case of the derivatives being dealt with – at least predominantly – are tetrahydo α-acid (THAA) or hexahydro- β-acid (HHBA), and in the case of the hops acid derivatives are iso-α-acid (IAA), rho-iso-α-acid (RIAA), tetrahydro-iso-α-acid (THIAA) and/or hexahydroxide-iso-α-acid, or in any event mixtures thereof.

9. Procedure according to one of the foregoing claims,

characterized by the fact that

provided as an alkaline medium is an alkaline hydroxide, in particular potassium hydroxide or sodium hydroxide, or a mixture thereof.

10. Procedure according to Claim 9,

characterized by the fact that

the concentration of the alkaline medium amounts to 0.1 - 5%, preferably 1 – 5% preferably 2 – 4% alkaline hydroxide.

11. Procedure according to Claim 1,

characterized by the fact that

besides the addition of the solution, supplied to the process medium is additionally alkaline lye.

12. Procedure according to one of the foregoing claims,

characterized by the fact that

the hops acid is dissolved in the alkaline medium as salt.

13. Procedure according to one of the foregoing claims,

characterized by the fact that

the solution is added to the process medium manually.

14. Procedure according to one of the foregoing claims 1 –12,

characterized by the fact that

the solution is added to the process medium over already available dosing systems.

15. Procedure for the production of a solution of hops acid for addition to a sugary, aqueous process medium, in particular of the sugar industry according to the procedure based on the foregoing claims 1 – 14,

the following procedural steps comprising:

a) preparation of an aqueous medium;
b) heating;
c) addition of hops acid, in particular melted hops acid, measuring the amount of hops acid such that the end concentration lies within a prescribed concentration range;
d) addition of the alkaline medium for reaching a predetermined pH value;
e) mixing the alkaline medium with the added-in hops acid;
f) maintaining the mixture at an elevated temperature over a prescribed period of time;
g) separating out the hops acid solution from the mixture or vice-versa, as well as
h) cooling the hops acid solution.

16. Procedure according to Claim 15,

characterized by the fact that

the concentration of the hops acid in solution lies in the range of 2 – 40%, preferably 5 – 20%, especially preferred 10 – 15%.

17. Procedure according to Claim 15 or 16,

characterized by the fact that

the mixture is held at a temperature in the range of 40 – 80° C, preferably 60 – 80° C, preferably 65 – 75° C.

18. Procedure according to the foregoing claims 15 – 17,

characterized by the fact that

hops acid solution is cooled down to a temperature below 10° C, preferably to a temperature in a range from 2 – 7° C.

19. Procedure according to one of the foregoing Claims 15 – 18,

characterized by the fact that

the separated out solution of hops acid displays a pH value in the range of 7.0 – 13.0, preferably 7.5 – 12.0, preferably 9.5 – 11.0.

20. Procedure according to one of the foregoing claims 15 – 19,

characterized by the fact that

used as hops acids are β-acids, α-acids, iso-α-acids or a mixture thereof, or isomerized hops acids and/or their derivatives, in particular –at least predominantly – tetrahydro-α-acid (THAA) or hexahydro-β-acid (HHBA) or iso-α-acid (IAA), rho-iso-α-acid (RIAA), tetrahydro-iso-α-acid (THIAA) and/or hexahydroxide-iso-α-acid, or a mixture thereof.

21. Use of hops acids for combating microorganisms in a sugary, aqueous process medium, in particular of the sugar industry,

characterized by the fact that

hops acid brought into an alkaline solution is added to the process medium, whereby the pH value of the solution is higher than the pH value of the process medium, and the hops acid in the process medium passes over from the dissociated form into the non-dissociated form based on one of the Claims 1 – 14.

WO00/53814 PCT/DE00/00485

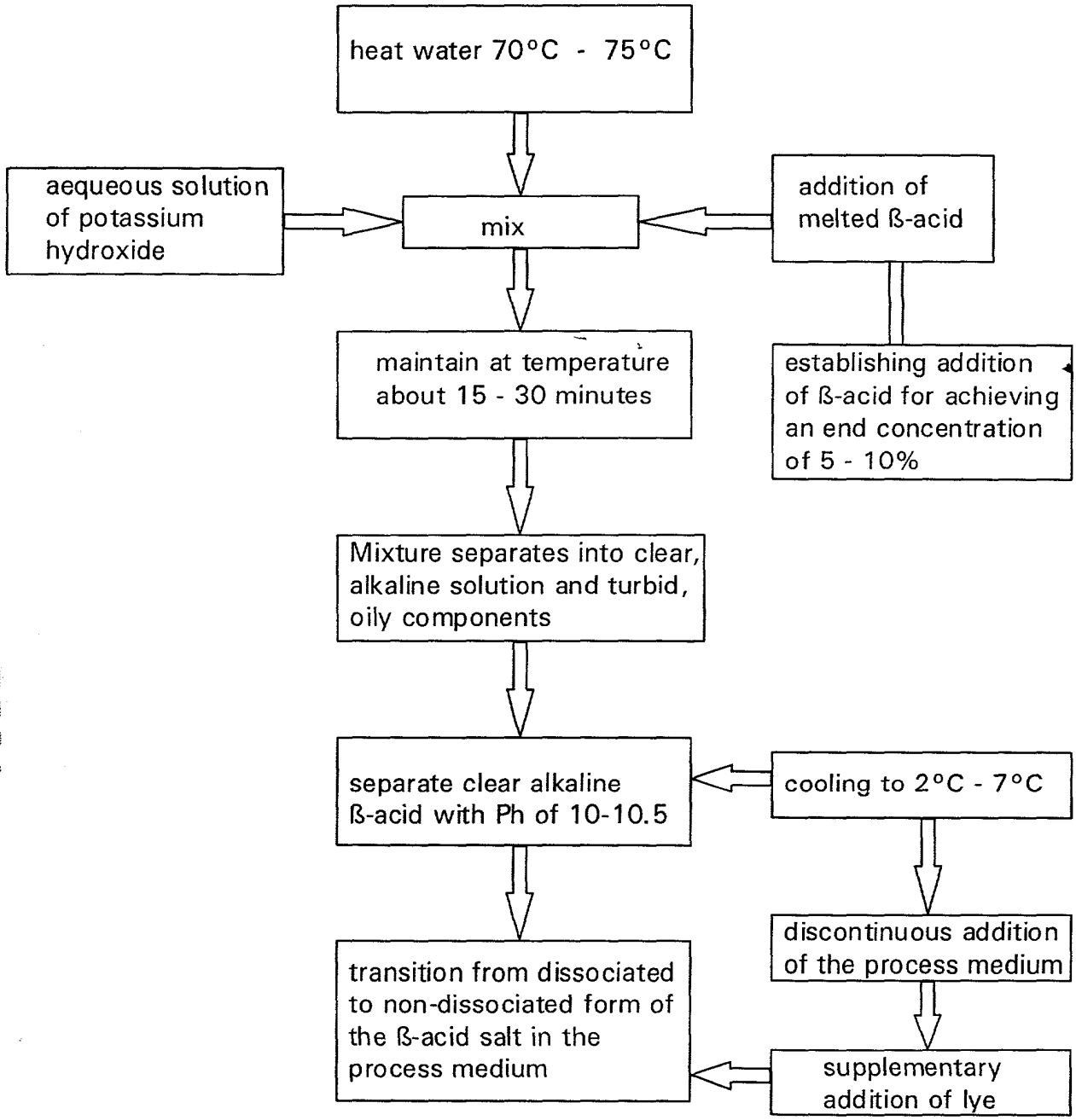

Fig. 1

Attorney Docket No. 11318.58USWO

MERCHANT & GOULD P.C.

United States Patent Application

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and sole inventor (if only one name is listed below) or a joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled: METHOD FOR CONTROLLING MICROORGANISMS IN A SUGAR-CONTAINING AQUEOUS PROCESS MEDIUM

The specification of which
a. ☐ is attached hereto
b. ☒ was filed on as application serial no. and was amended on (if applicable) (in the case of a PCT-filed application) described and claimed in international no. PCT/DE00/00485 filed February 22, 2000 and as amended on (if any), which I have reviewed and for which I solicit a United States patent.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I hereby claim foreign priority benefits under Title 35, United States Code, § 119/365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on the basis of which priority is claimed:

a. ☐ no such applications have been filed.
b. ☒ such applications have been filed as follows:

FOREIGN APPLICATION(S), IF ANY, CLAIMING PRIORITY UNDER 35 USC § 119			
COUNTRY	APPLICATION NUMBER	DATE OF FILING (day, month, year)	DATE OF ISSUE (day, month, year)
Germany	199 09 827.1	March 5, 1999	
ALL FOREIGN APPLICATION(S), IF ANY, FILED BEFORE THE PRIORITY APPLICATION(S)			
COUNTRY	APPLICATION NUMBER	DATE OF FILING (day, month, year)	DATE OF ISSUE (day, month, year)

I hereby claim the benefit under Title 35, United States Code, § 120/365 of any United States and PCT international application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, § 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. APPLICATION NUMBER	DATE OF FILING (day, month, year)	STATUS (patented, pending, abandoned)

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below:

U.S. PROVISIONAL APPLICATION NUMBER	DATE OF FILING (Day, Month, Year)

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199 09 827.1

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25/03/01	9/3/01
Ort, Datum	Ort, Datum
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I hereby appoint the following attorney(s) and/or patent agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith:

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I understand that the execution of this document, and the grant of a power of attorney, does not in itself establish an attorney-client relationship between the undersigned and the law firm Merchant & Gould P.C., or any of its attorneys.

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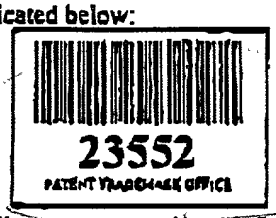

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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